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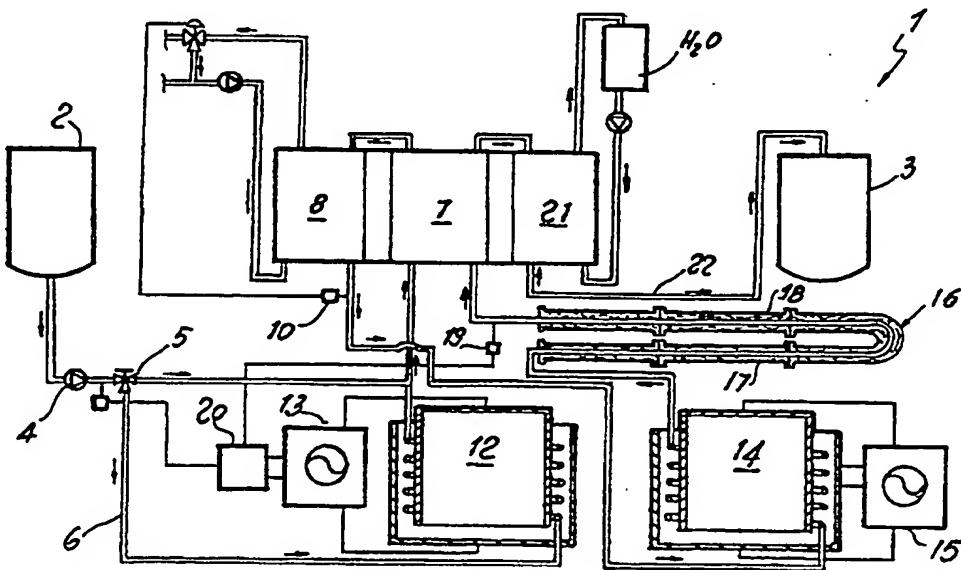
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(54) Title: METHOD AND APPARATUS FOR STERILIZING BIOLOGICAL LIQUIDS, PARTICULARLY MILK AND ITS BY-PRODUCTS



(57) Abstract

A method and apparatus for the continuous sterilization of unpackaged biological liquids, particularly milk and its by-products, containing bacterial and sporal loads, includes the steps of: a) gradually preheating the liquid with conventional heating methods to a temperature between 90° and 140 °C; b) irradiating the product with an electromagnetic RF field with a frequency of less than 1 GHz, so as to very quickly superheat the product to a temperature between 145 and 165 °C and with a thermal gradient between approximately 0.1 °C/s and 10 °C/s; c) cooling the liquid to a temperature of approximately 15 °C before packaging the product.

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METHOD AND APPARATUS FOR STERILIZING BIOLOGICAL LIQUIDS,  
PARTICULARLY MILK AND ITS BY-PRODUCTS

The present invention relates to a method for sterilizing  
5 biological liquids, for alimentary, sanitary, and cosmetic  
use, in particular milk and its by-products.

Various methods for preserving and sterilizing milk are  
known. It is known that the main components of this food  
10 product are constituted by water, sugars, mineral salts,  
fats, vitamins, biochemical compounds, such as enzymes,  
antibodies, and hormones, together with biological  
components of microbial and viral origin. Namely, milk is  
an emulsified solution of lactose, casein, fats, and some  
15 mineral salts in a water content of more than 85%. The  
pathogenic microorganisms that are present in this food  
product constitute the so-called bacterial flora, which  
includes, among others, *Mycrobacterium tuberculosis*, a wide  
range of viruses, including the poliomyelitis virus, as  
20 well as thermoresistant, thermophilic, psychrophilic and  
anaerobic germs.

The problem that must be solved by the sterilization of  
these products essentially consists in the total  
25 destruction of the bacterial flora without affecting the  
active principles that determine the original organoleptic,  
chemical and physical properties of the products allowing a  
shelf life comprised between 4 and 9 months depending on  
the national legislation.

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Conventional preservation methods used on an industrial scale are of the physical, chemical-physical, chemical, and biological types. In particular, physical methods use refrigeration, heat transmission, or desiccation.

5

Among these methods, processes based on the application of heat are by far the most effective and most widespread in solving the above mentioned problems. The heat application conditions depend not only on the nature of the product to be treated but also on the type of microorganisms contained in it and finally on the simultaneous use or not of other preservation processes. The degree of heat resistance of the microorganisms has to be linked to external and environmental factors, such as the initial microbial concentration of the medium, the characteristics of the medium itself, and the time and temperature parameters, as well as to intrinsic factors such as the heat resistance of germs and spores.

10  
15  
20  
25

In the conventional sterilization method, the product is superheating to temperatures of over 140°C for a time between 5 and 12 minutes.

The temperature and duration of the thermal treatment depend on the heat application method and on the type of product. Furthermore, at the end of the treatment the product must be subjected to cooling down to temperatures of less than 35°C before introducing it in sterilized containers (glass bottles or Tetra-pak<sup>R</sup> containers).

30

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Heat can be applied by indirect exchange, in which the product and the heating medium are separated by the wall of an exchanger, or by direct exchange, in which the product and the heating medium are in direct contact.

5

Current thermal preservation methods are the most important ones from the industrial point of view, but have some drawbacks.

10 In fact, in order to increase the sterilization effectiveness, it is necessary to increase the maximum temperature of the process, with the consequence of damaging the product from the organoleptic, physical and chemical point of view, destroying proteins and essential  
15 enzymes and giving it a cooked or burnt taste or reducing its natural taste and aroma.

Furthermore, in indirect-transmission systems the heat is transmitted from the outside inward, so that it is  
20 necessary to increase the temperature of the exchange surface in order to destroy the microorganisms even in the innermost regions. This can cause a partial non-uniformity and ineffectiveness of the process.

25 In direct-exchange systems, the heating medium is generally constituted by steam, which has the inconvenience of condensing inside the product itself, thereby reducing the concentration of the active principles; the steam must be eliminated before the condensation (degassing).

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Electronic preservation devices without an exchange surface have recently been devised which are based on the following principle.

5 Microorganisms, like all living organisms, are notoriously poor conductors of electricity and heat. Because of this, application of heat to these organisms is difficult and slow and furthermore occurs unevenly. In practice, due to their low electrical conductivity, microorganisms behave  
10 like dielectric particles that become aligned within an external electric field.

By using the above described physical principle, electronic preservation methods entail immersing the product in a  
15 high-frequency alternating electric or electromagnetic field for a period that is sufficient to cause the structural degeneration of the pathogenic microorganisms.

The Japanese patent application published as No. 2-211855,  
20 filed on February 10, 1989, describes a method and an apparatus for sterilizing a liquid food product by irradiation with high-frequency electromagnetic waves.

In this known method, the radiation is constituted by  
25 microwaves at frequencies above 1 GHz, which are emitted by a magnetron oscillator and are transmitted axially inside a waveguide, with very short irradiation times on the order of a few seconds. Due to the high frequency and short wavelength of the electromagnetic waves, shielding is  
30 necessary to protect the assigned personnel that works in

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the surrounding environment. Moreover, the intense superheating caused by the microwaves forces to perform very short treatments in quick succession, each treatment being followed by intense cooling to keep the product below  
5 the temperature at which its organoleptic chemical and physical properties change.

Patents US-A-2 576 862, US-A-3 272 636, FR-A-2 547 732, and DE-A-2 628 234 describe other methods and apparatuses that  
10 use electromagnetic waves whose frequencies are in the microwave and/or radio-frequency range. These known methods and apparatuses are applied to already-packaged products and always require appropriate shielding against emissions that are harmful to the human body.

15

Furthermore, since the destructive action of the alternating electromagnetic field affects not only the pathogenic microorganisms but also the active principles that determine the organoleptic properties of the products  
20 to be preserved, these known methods and apparatuses cause a reduction of these organoleptic properties, reducing the value of the active principles.

There are also known methods and apparatuses for preserving  
25 edible liquids with electromagnetic waves, in the radiofrequency field, with or without a conventional pre-heating. However, the results of such methods are still unsatisfactory according to both the microbacteria and enzymatic tests.

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The aim of the present invention is to obviate the above drawbacks, providing a method for the continuous sterilization of non-packaged liquid food and non-food products that allows complete and uniform preservation in 5 high-reliability conditions, leaving the organic and organoleptic properties of the treated products unchanged.

This aim, these objects, and others which will become apparent hereinafter are achieved by a method according to 10 claim 1 and an apparatus according to claim 6.

The method and the apparatus according to the invention provide for a particularly effective sterilization because of the radiofrequency irradiation in one or more passages 15 only during the high temperature treatment and for a very short time, preventing the destruction or alteration of the active principles and of the organic and organoleptic properties of the products.

20 Further characteristics and advantages will become apparent from the detailed description of a method and a device according to the invention, illustrated only by way of non-limitative example in the accompanying drawings, wherein:

25 Fig. 1 is a schematic view of a preferred embodiment of a sterilization apparatus according to the invention;

Fig. 2 is a view of a detail of the apparatus of Fig. 1, according to a second preferred embodiment of the 30 invention.

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With reference to Figure 1, this figure shows an apparatus for continuous sterilization according to the invention, generally designated by the reference numeral 1.

5 This apparatus includes a reservoir 2, made for example of stainless steel, for storing the product to be treated; the reservoir is connected to a vessel 3 that is similar to the preceding one, for collecting the treated product by virtue of a hydraulic circuit. A variable-delivery pump 4 is  
10 arranged in series to the reservoir 2, with a shunt valve 5 which conveys the product contained in the reservoir 2 through the pipe 6 of the circuit toward the primary circuit of a preheater 7, for example of the plate countercurrent type, which uses the superheated product  
15 itself as heating fluid.

Downstream of the preheater 7 there is a superheater 8 that uses a heating fluid - for example pressurized water at a temperature of approximately 150°C, that is contained in an  
20 external circuit 9 that is provided with a controlled bypass valve 10, or, alternatively, saturated steam directly injected in the product to be treated. At the output of the superheater 8 there is a temperature sensor 11 that controls the valve 10 so as to keep the temperature  
25 of the product between 90°C and 140°C, and preferably at about 130°C.

Downstream of the superheater 8 there is a first irradiation section, generally designated by the reference numeral 12,  
30 of the open type, for example having cylindrical symmetry,

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of the type described in Italian patent no. 1247067, so as to perform extremely uniform RF (radio-frequency) heating, for example up to a maximum temperature of approximately 165°C, for a relatively short time between for example 3 and 20 seconds, preferably for about 13 seconds.

Alternatively, the irradiation section may have a planar symmetry, of the type described in the cited Italian patent, and constituted by a pair of planar and parallel sheets and by a tubing interposed therein and made of a dielectric material in the shape of a coil wherein the product flows, as schematically illustrated in Fig. 2.

The cylindrical and coaxial plates of the irradiation section 12 are connected to a triode oscillator 13 or the like, set to generate radio waves at a frequency between 6 and 915 MHz, for example of about 27 MHz. The power of the electromagnetic field is selected in a range between 0.01 KW/l ( $10\text{KW}/\text{m}^3$ ) and 30 KW/l ( $3000\text{ KW}/\text{m}^3$ ) according to the composition of the product and to its bacterial concentration, measured beforehand by sampling bacterial swabs.

Because of the relatively high value of the wavelength of the radio waves emitted by the irradiation section, the apparatus does not require specific shielding for environmental safety, with the consequence of limiting the complexity and cost of the apparatus and of allowing its use in conventional installations without risks for the assigned personnel.

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The product that leaves the irradiation section 12 is conveyed to the secondary circuit of the preheater 7, or of a steam generator, not illustrated in the drawings, so as to recover part of the irradiation heat.

5

Then the product that leaves the preheater 7 is sent to a second irradiation section 14 that is similar to the preceding one but is connected to a second triode generator 15 or the like that is suitable to generate radio waves at 10 a substantially different frequency with respect to the first generator, for example a frequency that is about twice as high, so as to perform the RF sterilization of the residual spores.

15 The product that leaves the second irradiation section is degassed by eliminating the steam possibly injected during the pre-heating and then sent to an irradiated product holding region, with outgoing branches 17 and return branches 18 at a substantially constant temperature to 20 allow completion of the process for destroying the bacterial loads.

At the output of the portion 18 of the holding region 16 there is a temperature sensor 19 that sends an electrical 25 signal to a control unit 20 for controlling the oscillator 13 so as to keep the final temperature of the irradiated product below 165°C. An auxiliary cooling unit, not shown in the drawings, may furthermore be optionally provided between each turn of the dielectric duct of the irradiation 30 section, to ensure that the maximum preset temperature is

- 10 -

never exceeded.

At the output of the holding section 16, the fluid is fed into the secondary circuit of the preheater 7, which it 5 leaves at approximately 30-35°C, and then is fed into the main circuit of a refrigeration unit 21 that uses a mixture of water and glycol, kept at approximately 0°C by an appropriate chiller, as coolant. The product, cooled to a final temperature of approximately 15°C, is fed through the 10 pipe portion 22 toward the collecting container 3, from which it is removed to be packaged in adapted containers or aseptic packages.

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CLAIMS

1. A method for the continuous sterilization of non-packaged biological liquids, particularly milk and its by-products, containing bacterial and sporal loads,  
5 characterized in that it comprises the steps of:  
a) gradually preheating the liquid with conventional direct or indirect heating methods to a temperature between 90° and 140°C;  
b) irradiating the product with an electromagnetic RF field  
10 with a frequency of less than 1 GHz, so as to superheat the product to a temperature proximate to about 165°C and with a thermal gradient between about 0.1°C/s and 10°C/s;  
c) rapidly cooling the liquid to a temperature of approximately 15°C before packaging the product.

15

2. Method according to claim 1, characterized in that it comprises, after the RF irradiation step b), a step for cooling to a temperature that is equal to the preheating temperature, followed by a second step for irradiation with  
20 a RF whose frequency is different from that of step b).

3. Method according to claim 2, characterized in that the RF of the second irradiation step is about twice as high as that of the first step.

25

4. Method according to claim 1, characterized in that the frequency of the RF is between 6 MHz and 915 MHz and is preferably between 13 MHz and 96 MHz for milk and its by-products.

30

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5. Method according to claim 1, characterized in that the specific energy of the electromagnetic field is selected in a range comprised between 0.01 kW/l (10 kW/m<sup>3</sup>) and 30 kW/l (3000 kW/m<sup>3</sup>).

5

6. Apparatus for the continuous sterilization of biological and food products containing bacterial and sporal loads, particularly in the liquid or viscous state, comprising: a circuit that is suitable to connect a reservoir for storing  
10 the products to be treated to a container for collecting the treated products; a section for the conventional type preheating and superheating of the product to a temperature that is between 90° and 140°C; a means for irradiating the product with an oscillating high-frequency field; a means  
15 for confining a given amount of product within the field; and a means for adjusting the power and frequency of the electromagnetic field and the irradiation time as a function of the composition and concentration of the bacterial loads; characterized in that said irradiation  
20 means comprises at least a first RF heating section that is connected to the terminals of an oscillator that operates in the range of radio-frequencies below 1 GHz and is adjusted so as to superheat the product to a temperature proximate to 165°C; a thermally insulated region being  
25 provided downstream of said irradiation means to hold the product at the maximum temperature of the treatment and for a preset time that is sufficient to ensure complete destruction of the bacterial and sporal loads contained in the product, followed by a cooling means regulated to  
30 rapidly cool the treated products at a temperature of about

15°C before packaging.

7. An apparatus, according to claim 6, characterized in that it comprises a second irradiation section downstream of the first irradiation section and connected to the ends of an electromagnetic wave generator operating at a frequency which is different to said first frequency.
8. An apparatus, according to claim 7, characterized in that the frequency of the electromagnetic wave generator connected to said second irradiation section is about double that of the first frequency.
9. Apparatus according to claim 6, characterized in that said irradiation section comprises a pair of facing emitting surfaces forming between them at least one open interspace wherein said product confining means is arranged constituted by a tubing of dielectric material.
10. An apparatus according to claim 6, characterized in that said conventional preheating section comprises an heat exchanger or a saturated steam injector adapted to preheat the product to be irradiated, and using as heating fluid the product itself exiting the respective irradiation section.

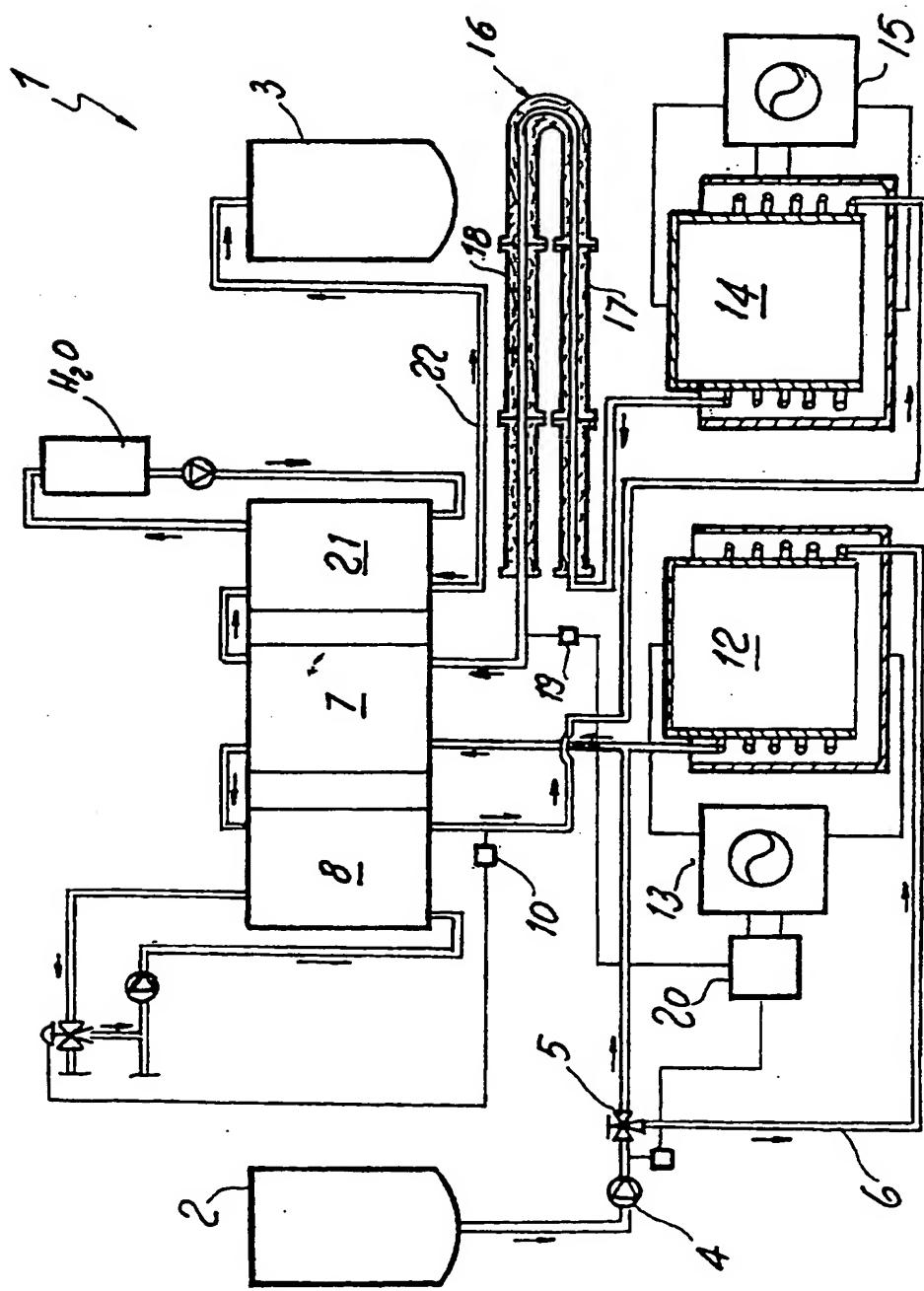


Fig. 1

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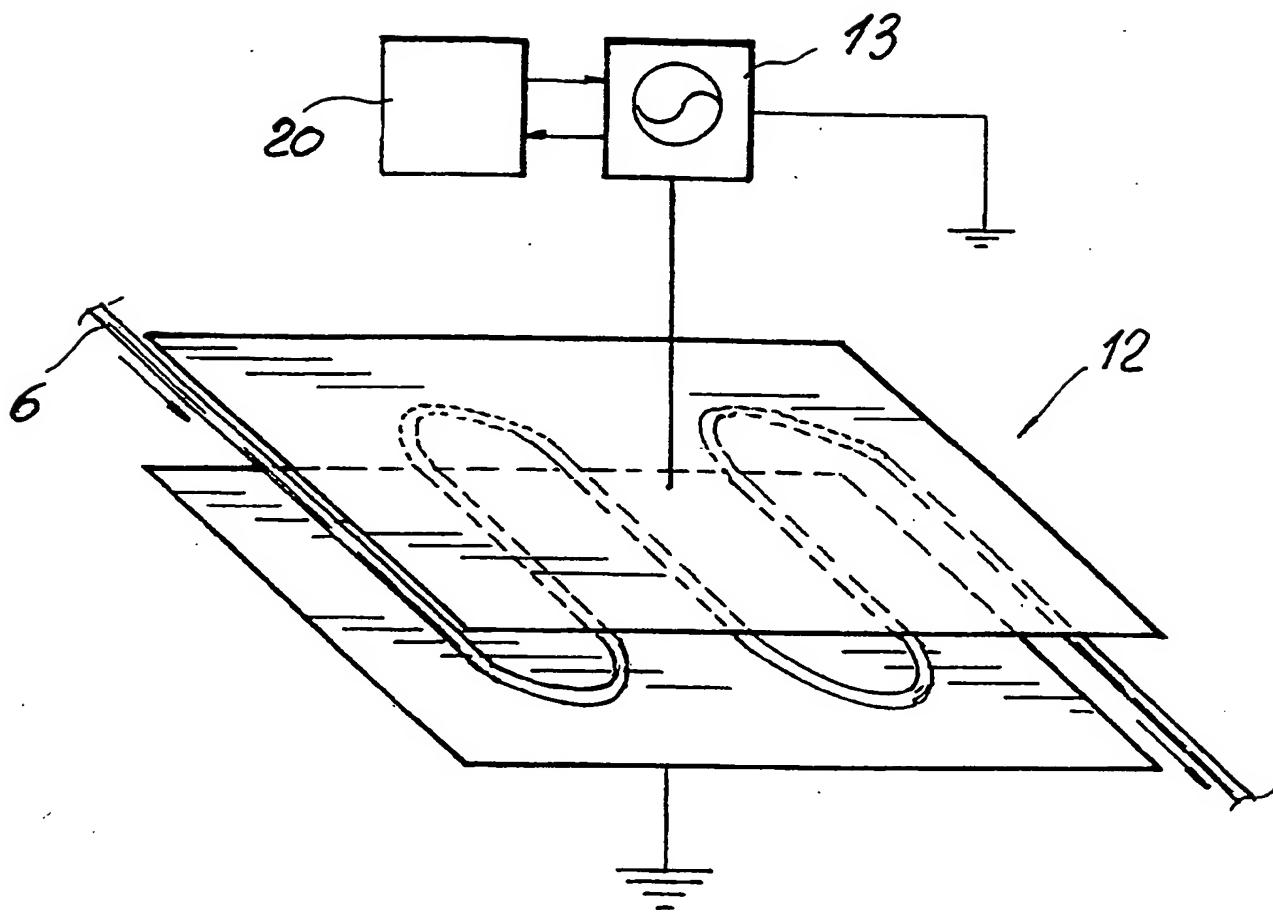


Fig. 2

## INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 96/02217

A. CLASSIFICATION OF SUBJECT MATTER  
 IPC 6 A23C3/07 A23L3/01 A61L2/12

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
 IPC 6 A23C A23L A61L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP,A,0 497 099 (OFFICINI DI CARTIGLIANO ) 5 August 1992 cited in the application see column 4 - column 9; claims 1-12; figure 6 & IT,A,1 247 067 ---	1-10
X	GB,A,619 495 (RADIO CORPORATION OF AMERICA) 10 March 1949 see page 4, line 5 - line 64; claims 1-7; figure 1 ---	1-10 -/-

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

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## INTERNATIONAL SEARCH REPORT

International Application No  
PCT/EP 96/02217

C(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	MILCHWISSENSCHAFT, vol. 41, no. 6, 1986, MUNCHEN DE, pages 337-339, XP002013016 G. PATIL ET AL.: "Deposit formation in UHT plants. I. Effects of forewarming in indirectly heated plants." see page 377 ---	1-10
A	WO,A,95 00179 (CHARM SCIENCES) 5 January 1995 see page 4, line 25 - line 33; claims 1,25; figure 5 ---	1,6
A	GB,A,682 064 (J. NYROP) 5 November 1952 see page 2, column 1; examples 1-6 ---	1
A	GB,A,2 274 568 (T. POLNY) 27 July 1994 see page 16 - page 21; claim 1; figure 1 ---	1,6
A	PATENT ABSTRACTS OF JAPAN vol. 16, no. 387 (C-0975) & JP,A,04 126060 (TOPPAN PRINTING CO), 27 April 1992, see abstract ---	1,6
A	PATENT ABSTRACTS OF JAPAN vol. 17, no. 394 (C-1088) & JP,A,05 076327 (MITSUBISHI KAKOKI KAISHA), 30 March 1993, see abstract ---	1,6,10
A	LU,A,55 053 (UNILEVER) 27 August 1968 see page 3 - page 4; claims 1-6; figure 1; examples 1-5 ---	1
A	GB,A,2 193 624 (APV INTERNATIONAL) 17 February 1988 see page 1, line 99 - page 2, line 53; claims 1-14; figure ---	1,6
A	US,A,5 403 564 (H. KATSCHNIG ET AL.) 4 April 1995 see column 3 - column 4; claims 1-13; figure 1 ---	1,6,10
A	EP,A,0 217 662 (S. CHARM) 8 April 1987 -----	

## INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 96/02217

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
EP-A-497099	05-08-92	CA-A- 2073609 IT-B- 1247067 US-A- 5288471 AT-T- 128821 DE-D- 69205296 DE-T- 69205296 ES-T- 2080966		11-01-94 12-12-94 22-02-94 15-10-95 16-11-95 05-06-96 16-02-96
GB-A-619495		NONE		
WO-A-9500179	05-01-95	US-A- 5389335 EP-A- 0703792 US-A- 5539673		14-02-95 03-04-96 23-07-96
GB-A-682064		NONE		
GB-A-2274568	27-07-94	CA-A- 2112315 DE-A- 4401735		23-07-94 08-12-94
LU-A-55053	27-08-68	BE-A- 707757 DE-A- 1692220 FR-A- 1548827 GB-A- 1187766 NL-A- 6716767 SE-B- 354566		10-06-68 29-07-71 06-12-68 15-04-70 10-06-68 19-03-73
GB-A-2193624	17-02-88	NONE		
US-A-5403564	04-04-95	AT-B- 399658 AT-A- 88493		26-06-95 15-11-94
EP-A-217662	08-04-87	CA-A- 1276563 DE-D- 3689580 DE-T- 3689580 JP-C- 1819456 JP-B- 5024794 JP-A- 62109572 US-A- 4839142 US-A- 4975246		20-11-90 10-03-94 11-05-94 27-01-94 08-04-93 20-05-87 13-06-89 04-12-90